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Program to Develop High Strength Aluminum Powder Metallurgy Products

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Phase IV A – Vacuum Process Verification

Reported by: W.S. Cebulak
Alcoa Research Laboratories
Physical Metallurgy Division

ALCOA

First Quarterly Report
June 20, 1972 to September 19, 1972

U.S. Army
Frankford Arsenal
Contract DAAA25-72-C0593

A Department of the Army Manufacturing Methods and Technology Project

October 12, 1972

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ALCOA

October 12, 1972

U. S. ARMY
FRANKFORD ARSENAL
CONTRACT DAAA25-72-C0593

PROGRAM TO DEVELOP HIGH STRENGTH ALUMINUM POWDER METALLURGY PRODUCTS

PHASE IVA - VACUUM PROCESS VERIFICATION

FIRST QUARTERLY REPORT

For the Period June 20, 1972
to September 19, 1972

W. S. Cebulak — 10-12-72
W. S. Cebulak
Project Engineer

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J. P. Lyle, Jr.
Project Supervisor

FOREWORD

This progress report was prepared for management purposes. It is a preliminary report of information generated during the first quarter of this investigation. The data and conclusions reported may be subject to major change. this report will be replaced by a summary report.

SYNOPSIS

Six P/M alloy-powders were prepared for this vacuum-preheat process verification. P/M 2" diameter extruded rod for extrusion evaluation was fabricated from vacuum preheated and hot pressed (VAC-preheat) compacts. Camparison ingot metallurgy (I/M) 7050 extrusions were also prepared.

Preliminary test results show P/M alloys MA86, MA87, MA89 and MA67 developed good transverse fracture toughness and ductility, with transverse fracture toughness comparable to that achieved in VAC-preheat MA83 alloy.[3] This verified the reproducibility of the fracture toughness improvement previously indicated for VAC-preheat over inert-gas (FCF) preheat.

P/M 4" diameter extruded stock for die forging was fabricated from VAC-preheated 140-lb compacts. Comparison I/M 7050 extruded rod was also prepared. This material is to be die forged during the second quarter.

Planned work in the second quarter includes evaluation of strength and fracture toughness of extrusions and die forgings and initiation of accelerated SCC testing to verify achieving the desired combinations of properties.

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INTRODUCTION

This program to continue the scale-up of high-strength aluminum powder metallurgy alloy products is a continuation of a program conducted as Phases I-III,[1,2,3] under Contract DAAA25-70-C0358. Efforts on these earlier phases have resulted in optimization and a first scale-up (to 170-lb compacts) of a P/M process that yields high-quality products which meet SNT Class A Airframe Ultrasonic Test Standards. Alloy optimization has resulted in the development of high-strength products with desirable combinations of engineering properties.

The original property goals of this program were to develop wrought products with the combinations of properties shown in Table 1. In extrusions from compacts preheated in flowing argon

Table 1

PROGRAM OBJECTIVES

Property	Combination A	Combination B	Combination C
Yield Strength (ksi)	95	85	75
K_{Ic} (ksi$\sqrt{\text{in.}}$)	26	26	45
SCC - ksi Sustained Stress	25	25	42
Fatigue Limit[1] - ksi	14	14	16
Exfoliation	High Resistance	Immune	Immune
Elongation (%)	11	11	11

Note: 1. Notched ($K_T = 3$), axial stress fatigue ($R = 0.0$), 10^8 cycles.

in an atmosphere furnace (FCE-preheat), MA66 alloy met the Target B properties[3] (Table 2). Against Target A, alloys MA67 and MA66

Table 2

PROPERTY GOALS OF TARGET B COMPARED TO MA66 ALLOY EXTRUSIONS

	Target	MA66 Alloy[4]
Y.S. - ksi	85	84.2
K_{Ic} - ksi$\sqrt{in.}$	26	28[1]
SCC - ksi Sustained Stress	25	25
Fatigue Limit[2] - ksi $K_t = 3$, $R = 0.0$	14	18.5[3]
Exfoliation	Immune	Resistant
Elongation - %	11	11.2

Notes: 1. Approximate K_{Ic} based on NTS/YS to K_{Ic} correlation.
2. Axial stress, 10^8 cycles.
3. In test, intact at 31.0×10^6 cycles.
4. Al-8.0 Zn-2.5 Mg-1.0 Cu.

Table 3

PROPERTY GOALS OF TARGET A COMPARED TO MA67 AND MA66 ALLOY EXTRUSIONS

	Target	MA67 Alloy[3]	MA66 Alloy[4]
Y.S. - ksi	95	95.9	94.3
K_{Ic} - ksi$\sqrt{in.}$	26	17[1]	26[1]
SCC - ksi Sustained Stress	25	25	<25
Fatigue Limit[2] - ksi $K_t = 3$, $R = 0.0$	14	20	20
Exfoliation	Resistant	Resistant	Resistant
Elongation - %	11	7.8	8.0

Notes: 1. Approximate K_{Ic} based on NTS/YS to K_{Ic} correlation.
2. Axial stress, 10^8 cycles.
3. Al-8.0 Zn-2.5 Mg-1.0 Cu-1.6 Co.
4. Al-8.0 Zn-2.5 Mg-1.0 Cu.

from FCE-preheated compacts closely approached the desired combination of properties, falling short on fracture toughness or SCC resistance, respectively,[3] and ductility (Table 3).

The Target C properties with the exception of fracture toughness have been met with extrusions from FCE-preheated compacts[2] (Table 4).

Table 4

PROPERTIES AT TARGET C

	Target C	Phase II Alloy[2]
Y.S. - ksi	75	75.2
K_{Ic} - ksi$\sqrt{in.}$	45	33[3]
SCC - ksi Sustained Stress	42	40
Fatigue Limit[1] - ksi	22	25
Exfoliation	Immune	Immune
Elongation - %	11	15

Notes: 1. Endurance limit for smooth specimen, rotating beam test.
2. Al-5.5 Zn-2.0 Mg-2.0 Cu.
3. Approximate K_{Ic} based on NTS/YS to K_{Ic} correlation.

Late in the planned period for Phase III, a new vacuum preheat/hot press (VAC-preheat) procedure was tried to confirm trends observed of the effects of gas content and porosity on fracture toughness. The effect of this procedure was a dramatic improvement in fracture toughness of P/M extrusions, notably in the transverse direction[3] (Fig. 1).

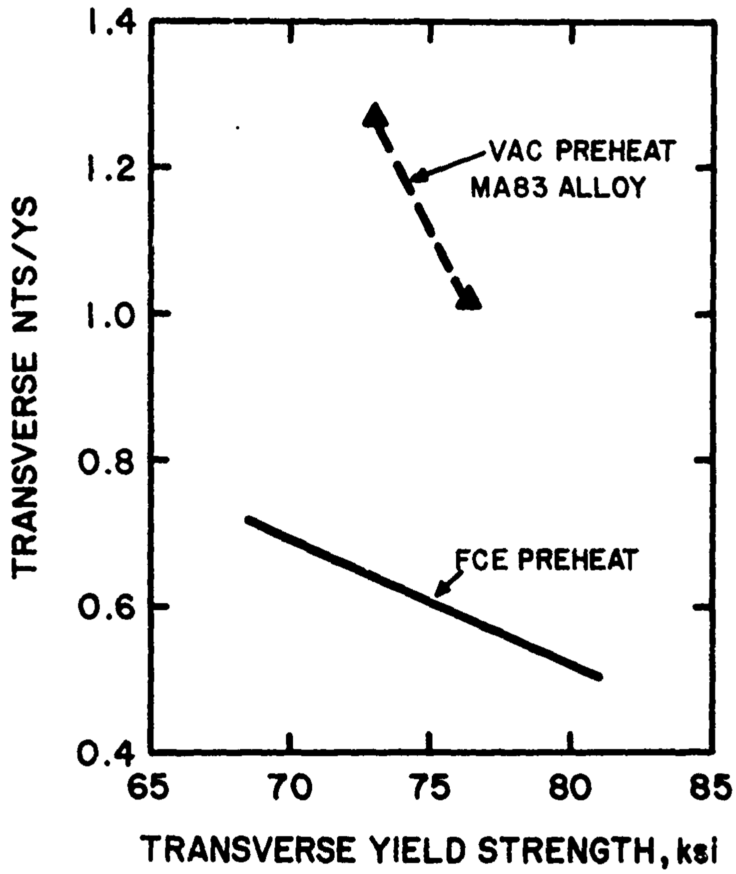

Fig. 1. Effect of VAC-preheat on the Transverse NTS/YS to Yield Strength Relation for P/M Extrusions Compared to FCE-preheat from Ref. 3.

Since this development occurred late in Phase III, it could not be incorporated in that material evaluation. Therefore, an experimental verification of the procedure and material testing against Targets A, B and C strength, SCC and fracture toughness goals were added at the beginning of Phase IV (scale-up to 3200-lb compacts) to determine if the primary goals of these Target properties could be achieved. The test products in the verification trial are extrusions and die forgings, while the alloys were selected on the basis of results achieved in Phases II and III.[2,3]

This first quarterly report of Phase IVA presents the detailed description of VAC-preheat material preparation for extrusions and die forging stock. Included are preliminary mechanical properties that verify the fracture toughness improvement with VAC-preheating.

OBJECTIVES

The objectives of Phase IVA are to verify the fracture toughness improvement with VAC-preheat over inert gas preheating and to achieve the following combinations of properties in extrusions:

Property	Target A	Target B	Target C
Longitudinal Yield Strength	95 ksi	85	75
Longitudinal K_{Ic}	26 ksi$\sqrt{in.}$	26	45
Transverse Sustained Stress without SCC	25 ksi	25	42

In die forgings, the yield strength goal differs from the above but the K_{Ic} and SCC goals are the same.

Table 5

COMPOSITION OF ALLOYS FOR VACUUM PREHEAT PROCESS VERIFICATION

Sample No.	Alloy	Powder Size[1] μM	Pot No.	Composition - Weight Per Cent Si	Fe	Cu	Mg	Zn	Be	Co	Zr	Ni
416237-2	MA86[2]	13.8	1647	.01	.01	1.99	2.31	5.69	.002			.00
416238-2	MA87[2]	14.1	1649	.05	.05	1.60[6]	2.42[6]	6.50	.004	.37[6]	.01[6]	.00
416239-3	MA88[2]	13.8	1648	.02	.02	2.29	2.52	6.50	.003		.13	.00
416240-2	MA89[3]	13.8	1650	.05	.07	1.08	2.35[6]	7.68[6]	.004[6]	.41		.01
416455-2	MA89[4]	13.4	1672	.03	.05	1.10	2.61	8.16	.003	.40		.01
416241-2	MA67[5]	14.4	1652	.06	.07	1.12	2.48	8.01	.003	1.36[6]		.02
416242-2	MA90[5]	13.4	1651	.04	.06	1.52	2.84	8.14[6]	.002	.41		.01
Ingot Metallurgy Control Material												
353758	7050[7]	Ingot[3]		.04	.04	2.41	2.20	6.21	.001		.10	

Notes:
1. Average Particle Diameter from Fisher Sub-Sieve Sizer.
2. Analytical Chemistry J.C. 72-073101.
3. Analytical Chemistry J.O. 72-073104.
4. Analytical Chemistry J.O. 72-090813.
5. Analytical Chemistry J.O. 72-080316.
6. Averages during atomizing--other elements do not appreciable change. Analytical Chemistry J.O. 72-072410.
7. Analytical Chemistry J.O. 72-031611. Also with 0.02% Ti.
8. Cast as 7" diameter D.C. ingot.

WSC:slo
10/10/72

PROGRESS IN THE FIRST QUARTER OF PHASE IVA

I. Two-inch Diameter Extrusions

A. Material Preparation. The alloys listed in Table 5 were prepared by melting and alloying in 1500-lb heats and air-atomizing to the powder sizes shown in Table 6. After scalping the powders

Table 6

POWDER SIZE AND SCREEN ANALYSIS FOR ALLOY-POWDERS FABRICATED IN VACUUM PREHEAT PROCESS VERIFICATION

Sample No.	Alloy	Pot No.	Date Atomized	Powder Size μm[1]	U.S. Standard Screen Analysis[2] -100, +200	-200, +325	-325	Scalping Screen (Tyler Mesh)
416237	MA86	1647	7-25-72	13.8	1.2	7.0	91.8	100
416238	MA87	1649	7-28-72	14.1	1.4	6.8	91.8	100
416239	MA88	1648	7-26-72	13.8	1.0	6.0	93.0	100
416240	MA89	1650	7-31-72	13.8	2.0	8.4	89.6	100
416455	MA89	1672	9- 6-72	13.4	1.0	6.0	93.0	100
416241	MA67	1652	8- 3-72	14.4	1.6	9.0	89.4	100
416242	MA90	1651	8- 2-72	13.4	1.6	7.6	90.8	100

Notes: 1. Average Particle Diameter from Fisher Sub-Sieve Sizer.
2. Analyses of drums numbered 1.

through a 100-mesh screen, 20-lb compacts were isostatically cold pressed at 38 ksi to approximately 80% density.

The 20-lb compacts listed in Table 7 were encapsulated in 6" O.D. x 1/4" wall x 13" long welded cans fabricated from extruded aluminum tube and plate. The cans had 1/2" aluminum pipe welded to one end for evacuation, while selected samples preheated with Ar or N_2 purge also had a 1/4" O.D. gas exit tube welded to the end opposite the evacuation line. Two cans were connected to a manifold which was in turn connected with flexible 2" I.D. vacuum hose to a Stokes Model 212H-10 mechanical vacuum pump. The compacts

Table 7

FABRICATING CONDITIONS FOR 20-LB. COMPACTS EXTRUDED TO 2" DIAMETER ROD

Extrusion Sample No.	Date Cold Pressed[1]	Date Extruded	Total Preheat Cycle hrs.	End of Preheat Vacuum[2] μM	Vacuum Gauge No.	Order Sealed[3]	Hot Compacting Pressure[4] ksi	Extrusion No.	Extrusion Breakout Pressure[5] ksi	Extrusion Length in.	Companion Piece No.[3]
MA86 Alloy: 5.7 Zn-2.3 Mg-2.0 Cu											
416237-1	8-17-72	8-30-72	3.7	5	1	First	156.6	8276	56.4	65.0	416240-2
416237-2	8-17-72	8-30-72	3.7	43	2	First	156.6	8274	71.4	64.5	None
416237-3	8-17-72	8-28-72	3.7	8	1	First	89.9	8258	56.4	66.0	416239-5
416237-4	8-17-72	8-29-72	3.5	50	2	First	32.0	8264	60.2	64.5	416241-5
416237-5	8-17-72	8-30-72	3.7[6]	8	1	First[6]	156.6	8279	52.6	65.5	None
MA87 Alloy: 6.5 Zn-2.4 Mg-1.6 Cu-0.4 Co											
416238-1	8-17-72	8-25-72	4.0	5	1	Second	156.6	8247	41.4	40.2	416240-5[10]
416238-2	8-17-72	8-28-72	3.6	70	2	First	156.6	8260	63.9	65.5	416241-2
416238-3	8-17-72	8-25-72	3.5	6	1	Second	156.6	8251	41.4	64.8	416238-4
416238-4	8-17-72	8-25-72	3.5	6	1	First	89.9	8250	37.6	64.8	416238-3
416238-5	8-17-72	8-29-72	3.9[6]	20	1	First[6]	156.6	8263	63.9	65.8	None
416238-6	8-17-72	8-29-72	4.1[7]	10	1	First[7]	156.6	8269	45.1	61.5	None
416238-7	8-17-72	8-30-72	3.8[8]	60	2	First[8]	156.6	8275	63.9	65.2	None
MA88 Alloy: 6.5 Zn-2.5 Mg-2.3 Cu-0.13 Zr											
416239-1	8-18-72	8-28-72	3.4	10	1	Second	156.6	8255	67.7	65.2	416242-1
416239-2	8-18-72	8-30-72	3.6	7	1	First	156.6	8272	74.4	64.8	416241-4
416239-3	8-18-72	8-29-72	3.6	48	2	First	156.6	8270	52.6	65.5	None
416239-4	8-18-72	8-30-72	3.6	38	2	First	89.9	8278	52.6	65.0	None
416239-5	8-18-72	8-28-72	3.7	8	1	Second	32.0	8259	60.2	66.0	416237-3
416239-6	8-18-72	8-31-72	3.5	44	2	First	156.6	8280	60.2		416239-7
416239-7	8-18-72	8-31-72	3.5	44	2	Second	156.6	8281	60.2		416239-6
MA89 Alloy: 7.7 Zn-2.4 Mg-1.1 Cu-0.4 Co											
416240-1	8-18-72	8-25-72	4.4	45	2	Second	156.6	8253	41.4	65.5	416240-3
416240-2	8-18-72	8-30-72	3.7	5	1	Second	156.6	8277	48.9	65.0	416237-1
416240-3	8-18-72	8-25-72	4.4	45	2	First	156.6	8252	41.4	66.0	416240-1
416240-4	8-18-72	8-25-72	3.8	50	2	First	89.9	8248	37.6	66.0	416242-2

(Continued on next page)

Table 7 (Continued)

FABRICATING CONDITIONS FOR 20-LB. COMPACTS EXTRUDED TO 2" DIAMETER ROD

Extrusion Sample No.	Date Cold Pressed[1]	Date Extruded	Total Preheat Cycle hrs.	End of Preheat Vacuum[2] μM	Vacuum Gauge No.	Order Sealed[3]	Hot Compacting Pressure[4] ksi	Extrusion No.	Extrusion Breakout Pressure[5] ksi	Extrusion Length in.	Companion Piece No.[3]
MA67 Alloy: 8.0 Zn-2.5 Mg-1.1 Cu-1.4 Co											
416241-1	8-18-72	8-28-72	3.6	90[9]	2	First	156.6	8257	63.9	64.0	416241-3
416241-2	8-18-72	8-28-72	3.7	70	2	Second	156.6	8261	60.2	64.8	416238-2
416241-3	8-18-72	8-28-72	3.6	90[9]	2	Second	156.6	8256	63.9	65.2	416241-1
416241-4	8-18-72	8-30-72	3.6	7	1	Second	89.9	827[illegible]	56.4	65.2	416239-2
416241-5	8-18-72	8-30-72	3.6	50	2	Second	32.0	8265	48.9	64.9	416237-4
416241-6	8-18-72	8-29-72	3.8	49[6]	2	First[6]	156.6	8271	45.1	64.5	None
416241-7	8-18-72	8-31-72	3.5	0.5	1	First	156.6	8282	52.6	64.5	None
MA90 Alloy: 8.1 Zn-2.8 Mg-1.5 Cu-0.4 Co											
416242-1	8-18-72	8-28-72	3.4	10	1	First	156.6	8254	67.7	64.5	416239-1
416242-2	8-18-72	8-25-72	3.8	50	2	Second	156.6	8249	37.6	65.0	416240-4
416242-3	8-18-72	8-29-72	3.7	4	1	First	156.6	8268	56.4	65.0	None
416242-4	8-18-72	8-29-72	3.6	14	1	First	89.9	8262	56.4	65.2	None
Ingot Metallurgy Control Material - 7050 Alloy											
353758-B	--	8-29-72	--	--	--	--	--	8266	(11)	186.0	--
353758-E	--	8-29-72	--	--	--	--	--	8267	(11)	176.0	--

Notes:
1. All 20-lb. compacts cold pressed at 38 ksi to approximately 5.2" dia. x 12.2" long.
2. All compacts preheated to 1000°F (see Figure 2 for heat up rate). Vacuum shown is Pirani Gauge Reading--to be calibrated against McLeod Gauge.
3. Two compacts connected to one pump with a manifold. Evacuation lines sealed in order shown.
4. Pressure maintained for 10 minutes, pressing compact against blind die in 6-3/8" diameter Extrusion Cylinder operated at 800°F.
5. Extrusion Ratio = 10.0 extruded at 0.5 ft/min. extrusion speed.
6. Preheated with ambient air in can, evacuated outside furnace immediately after removing from furnace.
7. Preheated with flowing (13 CFH) argon, removed from furnace, exit tube sealed and can evacuated immediately after removing from furnace.
8. Preheated with flowing (13 CFH) N_2, removed from furnace, exit tube sealed and can evacuated immediately after removing from furnace.
9. Evacuation line from manifold leak rod.
10. Dummy sample to measure heat-up rate, starting with cold furnace grate.
11. Not determined.

were preheated to 1000 F (heat up shown in Fig. 2) for the total time in the furnace shown in Table 7, with the vacuum pump operating

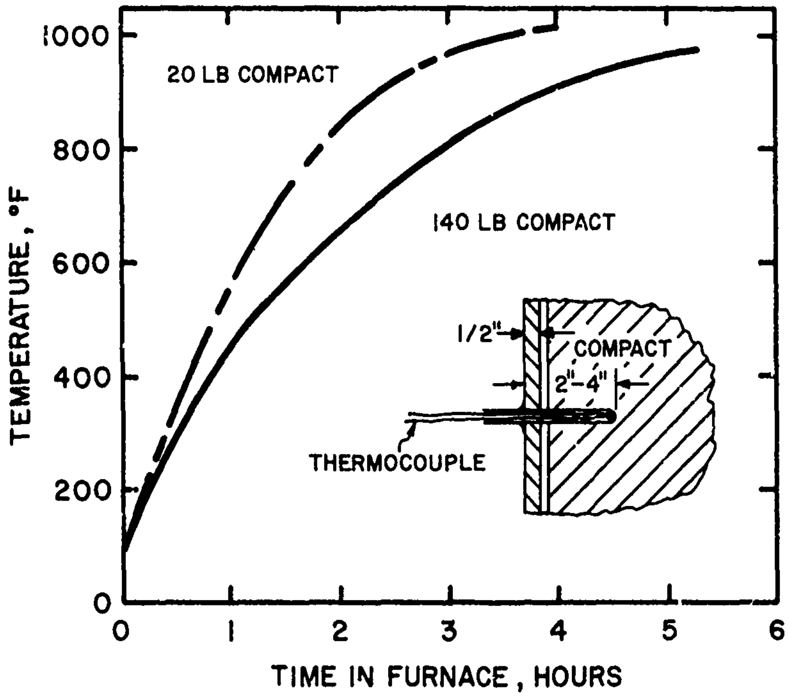

Fig. 2. Heat-up Rates of VAC-preheated Powder Compacts.

continuously during heat up. The pumps were pulling the vacuum through a dry ice-acetone cold trap and with the pump gas-ballast open except for the last 30 minutes of the preheat cycle. Selected samples were preheated with flowing Ar or N_2, or in a closed can (no circulating gas).

Upon completion of the heating cycle, the cans were removed from the furnace, the evacuation line was pinched closed and welded to retain the vacuum. Those samples preheated with no gas circulating were evacuated and sealed as above. The samples preheated with Ar or N_2 purge had their exit tubes welded closed, then were evacuated and sealed.

Immediately after sealing, the compact and can were hot pressed at 32 to 157 ksi for 10 minutes against a blind die (Table 7). This procedure constitutes the VAC-preheat. Immediately after hot pressing, the compact was extruded as 2" diameter rod from a 6-3/8" diameter extrusion cylinder, using fabricating conditions shown in Table 7.

These extrusions were ultrasonically inspected for defects and to determine the extent of the extrusion "pipe" defect, using a 10 MHz, 3/4" diameter lithium sulfate search unit, 3" water distance and 3.0" trace-to-peak indication from a 3-0075 Alcoa Series "D" ultrasonic standard reference block. The 2" diameter extrusions listed in Table 8 showed no isolated defects, very low ultrasonic noise levels and extrusion "pipe" to the extent shown.

Transverse notched tensile properties have been sensitive to the quality of the VAC-preheat. Accordingly, to verify the quality of the preheat, samples 3" long taken near the front of each extrusion (beginning 6" from the front of the extrusion) were solution heat treated for two hours, cold-water quenched and aged 24 hours @ 250 F + 4 hours @ 325 F as shown in Table 9. These sections were sampled for transverse notched tensile strength, with selected sections also being tested for longitudinal and transverse tensile properties.

Table 8

ULTRASONIC QUALITY OF THIRTY-SIX 2" DIAMETER EXTRUSION SECTIONS

Extrusion No.	Extrusion Length	Ultrasonic Noise Level[3] Maximum	Ultrasonic Noise Level[3] Minimum	Maximum % No. 3	Material to be Removed Due to Piping
416237-1	65"	.10	.05	3.3	18-3/4"
416237-2	64-1/2"	.10	.05	3.3	20-1/4"
416237-3	66"	.10	.05	3.3	20-1/2"
416237-4	64-1/2"	.10	.05	3.3	21-1/2"
416237-5	65-1/2"	.10	.05	3.3	23-1/4"
416238-1	46-1/4"	.10	.05	3.3	10-3/4"
416238-2	65-1/2"	.10	.05	3.3	20-1/4"
416238-3	64-3/4"	.10	.05	3.3	15"
416238-4	64-3/4"	.10	.05	3.3	17"
416238-5	65-3/4"	.10	.05	3.3	17-1/2"
416238-6	64-1/2"	.10	.05	3.3	24"
416238-7	65-1/4"	.05	--	2.5	23-1/2"
416239-1	65-1/4"	.10	.05	3.3	17-1/2"
416239-2	64-3/4"	.05	--	2.5	19-3/4"
416239-3	65-1/2"	.05	--	2.5	19"
416239-4	65"	.05	--	2.5	17-1/4"
416239-5	66"	.05	--	2.5	18-1/4"
353758-B1[1]	94"	.10	.05	3.3	0
353758-B2[2]	92"	.10	.05	3.3	15"
353758-E1[1]	84"	.10	.05	3.3	0
353758-E2[2]	92"	.10	.05	3.3	11-1/2"
416240-1	65-1/2"	.10	.05	3.3	15-1/2"
416240-2	65"	.05	--	2.5	19-1/2"
416240-3	66"	.05	--	2.5	20"
416240-4	66"	.05	--	2.5	19-1/2"
416241-1	64"	.10	.05	3.3	23-1/4"
416241-2	64-3/4"	.05	--	2.5	24-1/2"
416241-3	65-1/4"	.10	.05	3.3	18-1/2"
416241-4	65-1/4"	.10	.05	3.3	19"
416241-5	64-7/8"	.05	--	2.5	25"
416241-6	64-1/2"	.10	.05	3.3	18"
416241-7	64-1/2"	.05	--	2.5	18"
416242-1	64-1/2"	.10	.05	3.3	23-1/4"
416242-2	65"	.10	.05	3.3	20-1/4"
416242-3	65"	.05	--	2.5	20-1/2"
416242-4	65-1/4"	.05	--	2.5	18-1/4"

Notes: 1. Front half of extrusion.
2. Rear half of extrusion.
3. Inches on a 3" scale.
3" = 100% of No. 3.

WSC:slo
10/10/72

Table 9

EFFECT OF PROCESS VARIATIONS ON TRANSVERSE NOTCHED TENSILE STRENGTH OF 2" DIAMETER EXTRUDED P/M ROD

Extrusion Sample No.	Vacuum[3] μM	Hot Compacting Pressure ksi	Transverse Notched Tensile Strength[7] ksi
MA86 Alloy: 5.7 Zn-2.3 Mg-2.0 Cu[1]			
416237-1	5	156.6	90.8
416237-2	43	156.6	85.5
415237-3	8	89.9	74.3
416237-4	50	60.2[9]	87.1
416237-5	8[4]	156.6	50.7[8]
MA87 Alloy: 6.5 Zn-2.4 Mg-1.6 Cu-0.4 Co[2]			
416238-1	5	156.6	80.9
416238-2	70	156.6	82.0
416238-3	6	156.6	76.3
416238-4	6	89.9	81.1
416238-5	20[4]	156.6	79.0
416238-6	10[5]	156.6	81.6
416238-7	60[6,8]	156.6	48.5[8]
MA88 Alloy: 6.5 Zn-2.5 Mg-2.3 Cu-0.13 Zr[1]			
416239-1	10	156.6	66.8
416239-2	7	156.6	57.1
416239-3	48	156.6	61.8
416239-4	38	89.9	54.1
416239-5	8	60.2[9]	55.0
416239-6	44	156.6	59.3
416239-7	44	156.6	63.1
MA89 Alloy: 7.7 Zn-2.4 Mg-1.1 Cu-0.4 Co[2]			
416240-1	45	156.6	81.8
416240-2	5	156.6	81.6
416240-3	45	156.6	79.0
416240-4	50	89.9	76.7
MA67 Alloy: 8.0 Zn-2.5 Mg-1.1 Cu-1.4 Co[2]			
416241-1	90[8]	156.6	34.2
416241-2	70[8]	156.6	41.7
416241-3	90[8,11]	156.6	69.2
416241-4	7	89.9	70.2
416241-5	50	48.9[9]	67.6
416241-6	49[4]	156.6	68.5
416241-7	0.5	156.6	65.8
MA90 Alloy: 8.1 Zn-2.8 Mg-1.5 Cu-0.4 Co[1]			
416242-1	10	156.6	58.2
416242-2	50	156.6	52.2
416242-3	4	156.6	56.6
416242-4	14	89.9	50.4

Notes:
1. Solution heat treated 2 hours @ 910°F.
2. Solution heat treated 2 hours @ 920°F.
3. Pirani Gauge Readings--to be calibrated against a McLeod Gauge.
4. Preheated with no gas flow, then evacuated.
5. Preheated with flowing argon, then evacuated.
6. Preheated with flowing nitrogen, then evacuated.
7. 3" long piece from near the front of each extrusion solution heat treated, cold water quenched, aged 24 hours @ 250°F + 4 hours @ 325°F.
8. Suspected vacuum leak.
9. Extrusion Breakout Pressure - Hot pressed at 32 ksi for 10 minutes against a blind die before extruding.
10. M.T. 082872A, dated 9-5-72, 9-11-72.
11. This sample was the second compact sealed on a manifold; also connected to S-416241-1. The NTS suggests that the can on the first sample sealed was leaking.

B. Results and Discussion. The results of transverse notched tensile tests on 2" diameter extrusions are shown in Table 9. These results show some variability in notched tensile strengths between samples within an alloy that are principally associated with suspected vacuum leaks, in three samples of 34 extrusions prepared, or with planned variations in hot compacting pressure.

In four of the six alloys tested, marked improvements in transverse fracture toughness with VAC-preheat were indicated by the limited testing of the samples used for the preheat-quality check (Table 10) when compared to FCΔ (argon)-preheated extrusions from Phase III[3] (Fig. 3). These VAC-preheated extrusions in Phase IVA appeared to give comparable transverse fracture toughness to

Table 10

MECHANICAL PROPERTIES OF P/M ALLOY EXTRUSIONS[1]

Alloy	Sample No.	Longitudinal Properties T.S. ksi	Y.S. ksi	% El in 4D	R of A %	Transverse Properties T.S. ksi	Y.S. ksi	% El in 4D	R of A %	NTS ksi	NTS/YS
MA86	416237-1	87.0	79.0	12.5	26	77.8	68.2	11.0	14	90.8	1.33
MA87	416238-1	92.5	86.6	12.5	23	83.1	75.6	12.5	19	80.9	1.07
MA89	416240-1	90.5	86.8	12.5	32	82.8	76.8	8.5	12	81.8	1.06
MA67	416241-3	94.8	90.0	12.5	29	86.1	79.4	11.0	12	69.2	0.87

Notes: 1. Solution heat treated, cold water quenched, aged 24 hours @ 250°F + 4 hours @ 325°F.
2. M.T. 082872-A, dated 9-5-72, 9-11-72.

the VAC-preheated extrusion in Phase III, as shown in Fig. 3. This verifies the fracture toughness improvement with vacuum preheating and hot compacting.

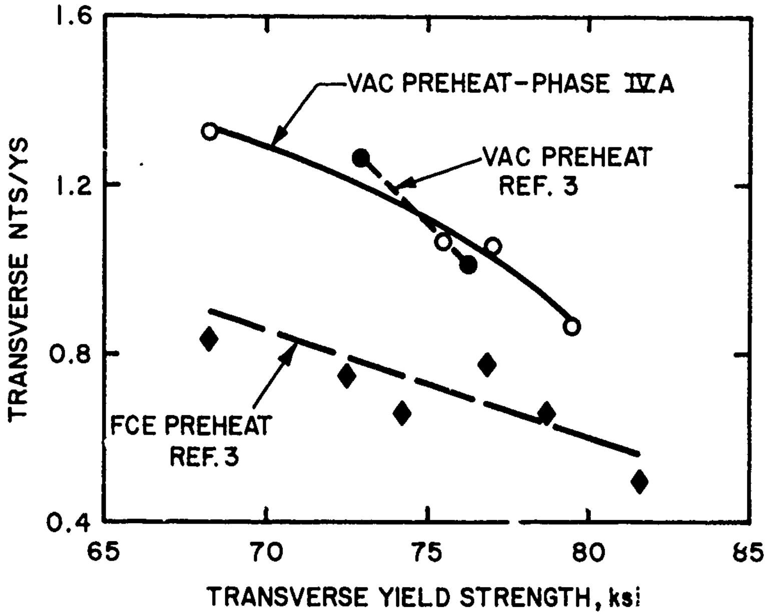

Fig. 3. Comparison of Transverse Fracture Toughness (NTS/YS) of P/M Extrusions from VAC-preheated Compacts (Phases III[3] and IVA) and FCE-preheated Compacts (Phase III[3]).

Alloys MA88 and MA90 appeared to develop somewhat lower transverse notched tensile strength (NTS) than the other alloys, including alloy MA67, with 1.6% Co (Table 9). Since these alloys have the highest Mg and Cu, the possibility of melting during solution heat treatment at 910 F was considered. Additional samples were heat treated at 880 F and aged as above, with the property results shown in Table 11. Reducing the heat treatment temperature slightly increased the NTS for MA88 alloy, but slightly decreased the NTS for MA90 alloy (Table 11). These differences are very slight and not significant, suggesting that melting may not

Table 11

EFFECT OF SOLUTION HEAT TREATMENT TEMPERATURE ON TRANSVERSE NOTCHED TENSILE STRENGTH OF MA88 AND MA90 EXTRUDED 2" DIAMETER ROD

Extrusion Sample No.	Vacuum[1] μM	Hot Compacting Pressure ksi	Transverse Notched Tensile Strength Solution Heat Treat Temperatures[2] 880°F ksi	910°F ksi
MA88 Alloy: 6.5 Zn-2.5 Mg-2.3 Cu-0.13 Zr				
416239-1	10	156.6	66.2	66.8
416239-2	7	156.6	61.2	57.1
416239-3	48	156.6	64.8	61.8
416239-4	38	89.9	60.8[3]	54.1
416239-5	8	32.0	61.0	55.0
416239-6	44	156.6	52.8[3]	59.3
416239-7	44	156.6	64.3	63.1
MA90 Alloy: 8.1 Zn-2.8 Mg-1.5 Cu-0.4 Co				
416242-1	10	156.6	53.6	58.2
416242-2	50	156.6	52.0[3]	52.2
416242-3	4	156.6	42.4	56.6
416242-4	14	89.9	52.0[3]	50.4

Notes:
1. Pirani Gauge Vacuum--to be calibrated against a McLeod Gauge.
2. Solution heat treated two hours, cold water quenched, aged 24 hours @ 250°F + 4 hours @ 325°F.
3. Single specimen. All others averages of two specimens.
4. M.T. 082872-A, dated 9-5-72, 9-11-72.

be the reason for low fracture toughness in these two alloys. These alloys will be examined further to determine if changes in heat treatment or aging will improve fracture toughness prior to testing these two alloys for SCC resistance and K_{Ic} over a range of yield strengths.

C. Conclusion. Vacuum preheating and hot compacting reproducibly improves the transverse fracture toughness of P/M extrusions.

II. Die Forgings - Preparation of Extruded Stock for Forging

A. Material Preparation. The alloy-powders listed in Tables 5 and 6 were prepared as described earlier. After scalping the powders through a 100-mesh screen, 140-142 lb-compacts listed in Table 12 were isostatically cold pressed at 30 ksi to approximately 80% density.

The compacts were encapsulated in welded aluminum cans fabricated from 8-1/8" O.D. x 1/4" wall x 42-1/2" long extruded tube and 1/2" end plates, with a 1/2" aluminum pipe welded to one end plate for evacuation. Each can was connected to a vacuum pump with a flexible 2" I.D. vacuum hose and evacuated prior to loading into a furnace.

The compacts were preheated to 950-1000 F (Heat-up shown in Fig. 2) for the total time in the furnace shown in Table 12, with the vacuum pump operating continuously during heat up. After the time in the furnace shown (when the vacuum achieved 70 μ or less), the cans were removed from the furnace, the evacuation pipe was pinched closed and sealed by welding to retain the vacuum. Each can and compact was then hot pressed in the cylinder shown in Fig. 4 with 32 to 90 ksi ram face pressure (see Table 12) for ten minutes, then ejected from the cylinder. This constitutes the VAC-preheat for large scale compacts. The relative sizes of the cold compact, the vacuum can and the hot pressed compact are shown in Fig. 5.

These 8.4-9.2" (tapered) diameter x 28" long hot pressed compacts were scalped to 7.5" diameter x 26" long to remove the can,

Table 12

PABRICATING CONDITIONS FOR 140-LB. COMPACTS EXTRUDED TO 4" DIAMETER ROD FOR DIE FORGING STOCK

Sample No.	Date Cold Pressed	Compact Length[1] in.	Date Hot Pressed	Total Preheat Cycle hrs.	End of Preheat Vacuum[2] μM	Vacuum Gauge No.	Hot Compacting No.	Hot Compact Pressure[3] ksi	Hot Compact Length in.	Extrusion No.[4]	Extrusion Breakout Pressure ksi
MA86 Alloy: 5.7 Zn-2.3 Mg-2.0 Cu											
416237-1	8- 9-72	40.3	8-23-72	4.8	28	1	8240	90.0	27.2	8304	60.7
416237-2	8- 9-72	41.4	8-21-72		210	3	--	--	--	--	--
416237-3	8- 9-72	41.4	8-22-72	4.4[5]	14[5]	1	8234	90.0	(13)	8311	46.4
MA87 Alloy: 6.5 Zn-2.4 Mg-1.6 Cu-0.4 Co											
416238-1	8-10-72	41.5	8-21-72	4.5	36	1	8227	90.0	(13)	8301	44.6
416238-2	8-10-72	41.5	8-22-72	4.3	22	1	8233	90.0	(13)	8305	48.2
416238-3	8-10-72	41.2	8-23-72	4.2	18	1	8243	90.0	27.7	8286	42.8[14]
416238-4	8-16-72	41.6	(10)							--	--
MA88 Alloy: 6.5 Zn-2.5 Mg-2.3 Cu-0.13 Zr											
416239-1	8-10-72	41.1	8-21-72	5.2	48	2	8231	90.0	(13)	8307	50.0
416239-2	8-10-72	41.7	8-21-72	4.6[6]	9[6]	1	8229	90.0	(13)	8296	46.4
416239-3	8-10-72	41.4	8-22-72	4.2	36	1	8235	90.0	(13)	8312	51.8
416239-4	8-10-72	41.4	8-23-72	4.2	60	2	8244	32.0[11]	27.5	8310	37.5
MA89 Alloy: 7.7 Zn-2.4 Mg-1.1 Cu-0.4 Co											
416240-1	8-11-72	40.7	8-22-72	4.3	60	2	8233	90.0	27.5	8300	42.9
416240-2	8-11-72	42.0	8-22-72	4.2[5]	14[5]	1	8237	90.0	(13)	8303	33.9
416240-3	8-11-72	41.4	8-23-72	4.2[5]	16[5]	1	8245	90.0	28.2	8306	50.0
MA67 Alloy: 8.0 Zn-2.5 Mg-1.1 Cu-1.4 Co											
416241-1	8-11-72	41.8	8-21-72	5.2	24	1	8233	90.0	(13)	8299	41.1
416241-2	8-11-72	41.9	8-21-72	4.6	60	2	8228	90.0	(13)	8309	42.7
416241-3	8-11-72	41.6	8-22-72	4.2	65	2	8236	90.0	(13)	8295	35.7
416241-4	8-11-72	41.6[7]	8-23-72	4.8[5]	21[5]	1	8242	32.0[12]	27.8	8298	48.2
416241-5	8-11-72	41.6	8-23-72	4.2	18	1	8246	90.0	(13)	(9)	--

(Continued on next page)

Table 12 (Continued)

FABRICATING CONDITIONS FOR 140-LB. COMPACTS EXTRUDED TO 4" DIAMETER ROD FOR DIE FORGING STOCK

Sample No.	Date Cold Pressed	Compact Length[1] in.	Date Hot Pressed	Total Preheat Cycle hrs.	End of Preheat Vacuum[2] µM	Vacuum Gauge No.	Hot Compacting No.	Hot Compact Pressure[3] ksi	Hot Compact Length in.	Extrusion No.[4]	Extrusion Breakout Pressure ksi
MA90 Alloy: 8.1 Zn-2.8 Mg-1.5 Cu-0.4 Co											
416242-1	8-11-72	41.6	8-22-72	4.1	28	1	8238	90.0	27.5	8302	41.1
416242-2	8-14-72	40.5	3-22-72	4.1	60	2	8239	90.0	27.1	8308	46.4
416242-3	8-14-72	41.6	8-23-72	4.8	60	2	8241	90.0	27.8	8297	46.4
Ingot Metallurgy Control Material - 7050 Alloy											
353758	--	--	--	--	--	-	--	--	--	8283	55.4
353758	--	--	--	--	--	-	--	--	--	8284	50.0
353758	--	--	--	--	--	-	--	--	--	8285	50.0

Notes:
1. Cold compacts approximately 7.4-7.5" diameter of 77-82% density. S-416237, 8 and 9 used 140-lb. powder charge; S-416240, 1 and 2 used 142-lb. powder. All isostatically cold pressed at 30 ksi pressure.
2. Pirani Gauge Reading--to be calibrated against McLeod Gauge.
3. Ram face pressure (maintained for 10 minutes) in cylinder shown in Figure 4, heated to 800°F.
4. Billets scalped to 7.5" diameter x 26" long (equal cuts off each end), induction reheated to 800°F, upset against a blind die in a 9-1/4" diameter extrusion cylinder operated at 800°F, and extruded at 0.5 ft/min. as 4" diameter rod. Extrusion Ratio = 5.4.
5. Preheated with pump capable of 220 µM vacuum, removed from furnace, evacuation line closed off with valve, can connected to vacuum pump No. 1 and evacuated to level shown.
6. Preheated with pump which only reached 160 µM, due apparently to water buildup in the pump oil. Disconnected pump (bleeding air into can), connected can to No. 1 pump, and evacuated to level shown.
7. Green compact end cracked while loading compact into can. 1-1/2" of compact affected.
8. Sample used for thermocouple dummy.
9. Sample was not scalped or extruded.
10. Retained as a cold pressed green compact.
11. After scalping, billet upset in extrusion cylinder at 68.8 ksi (no dwell).
12. After scalping, billet upset in extrusion cylinder at 69.6 ksi (no dwell).
13. Not measured.
14. Excessive lubricant caused extrusion skull as a skin on rod surface over trapped lubricant.

WSC:slo
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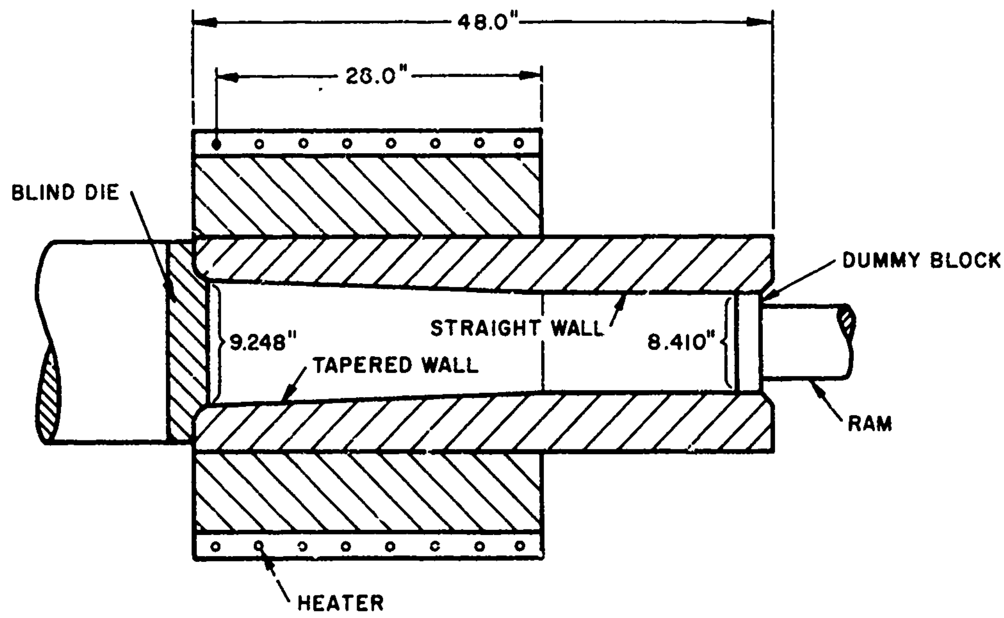

Fig. 4. Schematic of 8.4" Diameter Hot Compacting Cylinder.

induction reheated to 800 F, upset in a 9-1/4" diameter extrusion cylinder operated at 800 F and extruded as 4" diameter rod (extrusion ratio = 5.4) at 0.5 feet per minute under conditions shown in Table 12.

This material was shipped to Alcoa's Vernon Works to be die forged in Alcoa Die 9078, the same forging section evaluated in Phase III of this program.[3]

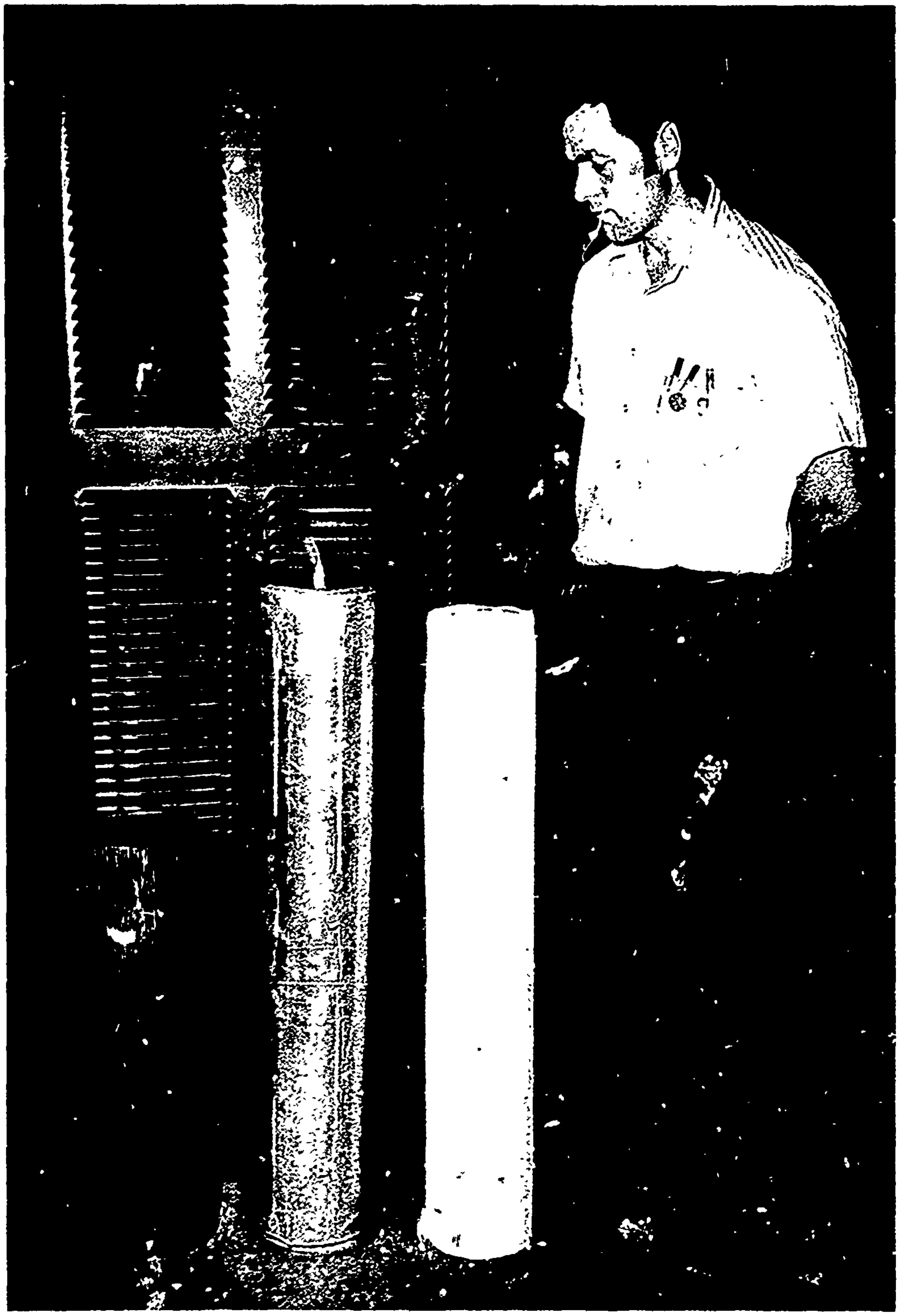

Fig. 5. Production Sequence of Hot-pressed P/M Compacts. From the right - Cold Compact, Canned Compact, Hot-pressed Compact.

WORK TO BE COMPLETED IN THE SECOND QUARTER OF
PHASE IVA, SEPTEMBER 20, 1972, THROUGH DECEMBER 20, 1972

1. Heat treat extrusions, determine tensile, fracture toughness, and accelerated stress-corrosion properties of 2" diameter extrusions in P/M alloys MA86, MA87, MA89, and MA67 and in I/M 7050 alloy in tempers aimed at target strengths.
2. Determine if changing thermal treatments can improve the fracture toughness of MA88 and MA90 alloy extrusions. Initiate mechanical property and stress-corrosion studies if toughness can be improved.
3. Heat treat, test 4" diameter rod for transverse notched tensile strength to assess quality of the VAC-preheat and determine the effect of the amount of reduction on fracture toughness.
4. Complete die forging from extruded P/M stock.
5. Heat treat die forgings, determine strength and fracture toughness. Initiate accelerated stress-corrosion tests of die forgings in P/M alloys MA86, MA87, MA89, and MA67 and in I/M 7050 alloy in tempers corresponding to those used on 2" diameter extruded rod.
6. Heat treat, test MA88 and MA90 die forgings on the basis of item 2 above.

REFERENCES

1. Cebulak, W. S., and Truax, D. J., "Program to Develop High-Strength Aluminum Powder Metallurgy Products - Phase I - Process Optimization," Alcoa Research Laboratories, Contract No. DAAA25-70-C0358 (Frankford Arsenal), March 12, 1971, AD 882-137L.

2. Cebulak, W. S., and Truax, D. J., "Program to Develop High-Strength Aluminum Powder Metallurgy Products - Phase II - P/M Alloy Optimization," Alcoa Research Laboratories, Contract No. DAAA25-70-C0358 (Frankford Arsenal), June 1, 1971, AD 884-642L.

3. Cebulak, W. S., and Truax, D. J., "Program to Develop High-Strength Aluminum Powder Metallurgy Products - Phase III - Scale-up A," Alcoa Research Laboratories, Contract No. DAAA25-70-C0358 (Frankford Arsenal), September 29, 1972.